

## PATENT SPECIFICATION

DRAWINGS ATTACHED

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## COMPLETE SPECIFICATION

## An Improved Process for Producing Activated Carbon

I, MINISTER OF AVIATION, formerly  
MINISTER OF SUPPLY, London, do hereby  
declare the invention, for which I pray that  
a patent may be granted to me, and the  
method by which it is to be performed, to  
be particularly described in and by the  
following statement:—

This invention relates to an improved pro-  
cess for producing activated carbon from  
carbonaceous material.

By means of the present process activated  
carbon is produced directly from carbon-  
aceous raw material, the only preparation of  
which for anthracite, for example, is size  
grading so that preparatory operations such  
as fine grinding, briquetting and pre-carbon-  
ising may be avoided.

In accordance with the invention, a process  
for producing activated carbon comprises  
heating a bed of granular carbonaceous  
material to a temperature between 800° and  
950° C. and subjecting the bed to a stream  
of hot activating gas preheated, before it  
enters the bed, to a temperature of at least  
800° C. and about that of the bed, the gas  
being arranged to flow upwards through  
the bed with a velocity sufficient to form  
a fluidised bed.

Preferably, granulated carbonaceous  
material ground to a particle size similar to  
that required in the product is supported in  
a furnace tube at temperatures between 800°  
and 950° C. and steam, used as activating  
gas, is blown up through the bed at a  
sufficient velocity to form a fluidised bed.

A preferred carbonaceous material such  
as anthracite does not give too high a pro-  
portion of volatile products and a batch  
process for producing homogeneous activated  
carbon from anthracite will now be described  
by way of example.

300 parts by weight of anthracite ground

14 to 22 B.S.S. are charged into a vertical  
reactor which comprises a silica tube which  
is surrounded by an electric furnace and  
which has a cross-gauze of stainless steel to  
support the ground bed of anthracite. With  
dried nitrogen flowing upwards through the  
bed the temperature of the reactor is raised  
rapidly to a temperature between 800° and  
950° C. At this temperature the nitrogen,  
if supplied, is replaced by steam at the rate  
of 15 parts by weight per minute and the  
steaming supply is continued at constant  
temperature until the required yield of  
activate is reached. The heat is then re-  
moved and the charge cooled, if desired, in  
a slow supply of nitrogen.

The activation reaction is carried out  
effectively by preheating the activating gas  
to a temperature of at least 800° C. and  
about that of the fluidised bed before it flows  
through the bed so that the temperature  
of the bed can be maintained within the re-  
quired temperature range throughout its  
height. A pronounced overall movement  
is then set up in the bed with ascending  
and descending streams of material.

It has been found that, at a given tem-  
perature within the required range from 800°  
to 950° C., the specific surface area, that  
is the internal surface area per unit weight  
of activate, produced by steam activation and  
the loss of weight increased with the duration  
of activation. Increasing activation also re-  
sulted in a decrease in bulk density of the  
activate. It has also been determined that  
the temperature of activation had a marked  
effect on the size of pores and the specific  
surface area; lower temperatures of 800° to  
850° C. favouring a higher surface area and  
minute pores suitable for gas absorption. The  
bulk density showed only a slight change  
with temperature; at a given yield (pro-

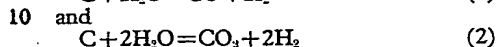
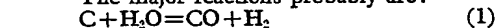
आई. पी. एम. डी. (सी.एस.आई.आर.)  
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portion of material by weight remaining after activation) the specific surface area decreased with increases of temperature. Over the temperature range 815° to 950° C. the rate of reaction increased six fold; but the lower reaction temperature yielded a more reactive product which is more highly gas absorbent.

The major reactions probably are:—



The amount of hydrogen evolved in test runs corresponded more nearly to reaction (1) than (2) as did the  $CO/CO_2$  ratio which was about 3.0 and varied a little with temperature. The total volume of gases produced was proportional to the loss in weight of the bed, and the measurement of the gas evolution formed a good indication of when to stop a reaction to obtain a given yield of activated carbon.

Results showed that at yields above 40% (by weight), less than 10% of the product passed 22 B.S.S., the proportion increasing with increasing activation. In experiments using anthracite closely graded to 14×16 mesh and giving a 40% yield, 84% of the activate remained on 18 B.S.S. and 97% on 22 B.S.S.; at 20% yield 90% remained on 22 B.S.S.

The specific surface area increased linearly with activation; at 70% yield i.e. the percentage weight remaining of the weight of carbonaceous starting material, the specific surface area was 110 m<sup>2</sup>/g and at 14% yield activation the specific surface area was 280 m<sup>2</sup>/g.

The bulk density of the activate fell approximately linearly with decreasing yield; at 80% yield, the density was 0.80 g/ml, at 20% yield, the density was 0.35 g/ml.

The density measured by methyl alcohol displacement rose with increasing activation: however, the absorption forces give rise to a compression effect proportional to the internal surface area, and on correcting for this, the true density was found to vary but little with the extent of activation.

Whereas in the above example anthracite has been used as the raw material, other carbonaceous materials such as semi-anthracite and dry steam coal may be successfully activated using a fluidised bed according to the invention. Other solid materials may require to be carbonised before activation either in the same reactor or separately; coco-nut shell, for example, may be successfully activated after pre-carbonisation.

Steam may be used at the start of the reaction in place of nitrogen which is used to displace air in the apparatus, and to "stir" the bed to obtain an initial uniform temperature distribution. Also a mixture which may include hydrogen and carbon monoxide from the activating reaction may

be burned in an auxiliary burner to attain the required high temperature and the burnt gases may be used directly to heat, and fluidise and activate the bed, or the heat may be transferred by conduction or convection, the bed being fluidised with a separate source of activating gas.

A suitable activating gas which may be employed is steam to which has been added air or carbon dioxide or a mixture of these with an activating temperature within the range 800—950° C.

Further to facilitate the transfer of heat to the gas stream an auxiliary fluidised bed containing, for example, alumina granules may be positioned below the bed of carbonaceous material.

The process for producing activated carbon may be advantageously operated as a continuous process which comprises heating a bed of granular carbonaceous material to a steady temperature between 800° and 950° C., continuously supply fresh carbonaceous material to the bed and continuously passing a strong stream of activating gas through a heat exchanger and thence upwards through the bed with a velocity sufficient to form a fluidised bed, and continuously withdrawing activated carbon from the top of the bed and spent gas which is passed through the heat exchanger, the activating gas being pre-heated, before it reaches the fluidised bed, to a temperature of at least 800° C. and about that of the bed.

A typical continuous process for producing activated carbon suitable for gas absorption will now be described, by way of example, with reference to the accompanying drawing which shows a cross-sectional side elevational view of a continuous reactor.

As shown in the drawing the reactor consists essentially of a long lagged metal tube 1, made for example of stainless steel, which forms a tubular furnace within which a bed of carbonaceous material 2 is supported by a gauze 3 above a steam generator 4. Fresh carbonaceous material is fed into the furnace from a hopper 5 through a valve 6 and the bed 2 is maintained at a constant height by means of an overflow tube 7 which descends through the centre of the main tube 1. The tube 7 may also have a controlling outlet valve 8 which like the valve 6, preferably is a type, such as a worm thread type, resistant to gas flow. A further outlet tube 9 is provided to remove the hot spent activating gases and is preferably coiled below the bed as shown to form a heat exchanger 10.

In operation, the bed 2 is heated up to the operating temperature between 800° and 950° C. by a surrounding furnace heater 11 and steam is produced from the water 12 at the base of the tube 1 by heat from the hot tube 9 and the heated lagged main tube

1. Replacement water is fed at a steady rate through a delivery pipe 13. The furnace heater 11 may conveniently be an electrical heater and an independent controlling water heater may be provided.

Steam is arranged to be generated with sufficient vigour for the bed to be fluidised as the steam rises through it and the gaseous products of the activating reaction to pass out through the tube 9. After a period of an hour or so, depending on the temperature, fresh carbonaceous material is admitted to the bed at a steady rate from the hopper 5 through the valve 6. This causes activated or partly activated material at the top of the bed to overflow into the tube 7 and be discharged through the valve 8. Ascending and descending columns of material are established substantially throughout the height of the fluidised bed (there generally being a rising central column with surrounding descending columns) and, as the carbonaceous material becomes lighter as it is activated, when correct operating conditions have been established, lighter activated carbon overflows into the tube 7; as heavier only partly activated carbon largely does not reach the extreme top of the bed during its circulation through the bed.

For effective operation the bed must not be too shallow and, as previously described, it is important for the activating gas to be as nearly as possible at the temperature of the bed on entry to enable the activating temperature and uniform activating conditions to be maintained throughout the height of the bed.

The required preheating of the activating gas may be effectively carried out by using the heat of the spent gases withdrawn from the hot fluidised bed in a heat exchanger together with the heat from the heater 11. In the reactor shown in the drawing, the gaseous output of the reactor passes down through the tube 9 and heats the stream of steam ascending from the generator 4 so that it is at or nearly at the activation temperature when it reaches the bed 2.

Typical operating conditions in a 10 inch diameter reactor having an 8—10 inch high bed containing 2—3 kg of anthracite being activated are as follows. Anthracite at a steady rate of about 1 kg/hour is supplied to the bed and water is converted into steam at a rate of about 6 kg/hour of which about 20% is utilised in the activation reaction. Depending upon the steaming rate and the temperature, the output rate of activated carbon is 350—450 g/hour. For producing activated carbon suitable for gas absorption a temperature around 850° C. is preferred, the activation time then being of the order of 2—3 hours.

#### WHAT I CLAIM IS:—

1. A process for producing activated carbon which comprises heating a bed of granular carbonaceous material to a temperature between 800° and 950° C. and subjecting the bed to a stream of hot activating gas preheated, before it enters the bed to a temperature of at least 800° C. and about that of the bed, the gas being arranged to flow upwards through the bed with a velocity sufficient to form a fluidised bed.

2. A process for producing activated carbon which comprises heating a bed of granular carbonaceous material to a temperature between 800° and 950° C., passing a stream of activating gas through a heat exchanger and thence upwards through the bed with a velocity sufficient to form a fluidised bed, and withdrawing the spent activating gas through the heat exchanger, the activating gas being preheated before it reaches the bed, to a temperature of at least 800° C. and about that of the bed.

3. A process according to Claim 1 or Claim 2 in which the carbonaceous material is an anthracite.

4. A process according to Claim 1, Claim 2 or Claim 3 in which the activating gas is steam.

5. A continuous process for producing activated carbon which comprises heating a bed of granular carbonaceous material to a steady temperature between 800° and 950° C., continuously supplying fresh carbonaceous material to the bed and continuously passing a strong stream of activating gas through a heat exchanger and thence upwards through the bed with a velocity sufficient to form a fluidised bed, and continuously withdrawing activated carbon from the top of the bed and spent gas which is passed through the heat exchanger, the activating gas being preheated, before it reaches the bed, to a temperature of at least 800° C. and about that of the bed.

6. A continuous process according to Claim 5 in which the spent gas is withdrawn downwards in a pipe through the bed and heats the rising stream of activating gas in the heat exchanger.

7. A continuous process according to either Claim 5 or Claim 6 in which the carbonaceous material is anthracite.

8. A continuous process according to Claim 5, Claim 6 or Claim 7 in which the activating gas is steam.

9. A continuous process for producing activated carbon substantially as hereinbefore described with reference to the accompanying drawing.

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COMPLETE SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale.

